

Developments in The Pressure Field At CEM

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Abstract

In the near future new pressure standards will be operative at CEM. These standards together with the existing will provide traceability to the whole pressure range. These standards are a mercury column, a force piston gauge and a static expansion system.

The mercury column is a U-shaped steel tube filled with mercury. Its measurement principle is Torricelli's law. The height difference between the two columns is measured by means of a single-beam helium–neon laser interferometric system. Its measurement range is from 1 kPa up to 130 kPa in absolute, relative and differential modes.

The force piston gauge is a recent acquisition. It is a pressure standard designed to cover the range from less than 1 Pa to 15 kPa gauge and absolute pressures. The instrument operates on the piston gauge principle in which the pressure on the piston is measured by a forced balanced load cell.

The static expansion system is based on Boyle's law with some corrections. This standard will work from 10⁻⁴ Pa to 103 Pa. This system consists of two large 100-litre volumes, two 1-litre volumes and a 0.5-litre volume. The system is evacuated by means of two 300 l/s-turbomolecular pumps connected to the large vessels. Both of them will be connected to a primary dry pump.

Keywords: mercury column, force piston gauge, static expansion system.

1. Mercury Column

1.1 Introduction

A mercury column laser interferometer system [1] has been designed at Centro Español de Metrología (CEM). Its measurement range is from 1 kPa up to 130 kPa in absolute, relative and differential modes. This standard will provide traceability to piston-cylinder assemblies calibration.

The measuring principle is based on Torricelli's experience. If different pressures are applied between the two branches of the U-shaped tube filled with a liquid with density ρ , its difference ΔP can be calculated according to:

$$\Delta P = \rho \cdot g \cdot \Delta h \quad (1)$$

where g is the local gravity acceleration.

In this design mercury is used as the liquid and nitrogen is used to generate pressure. Pressure P in the measurement branch is given by the following equation,

$$P = \rho_{Hg} \cdot g \cdot h - \rho_{N_2} \cdot g \cdot L + P_0 \quad (2)$$

where P_0 is the pressure at the reference branch, h is the height difference between mercury levels (reference branch minus measurement branch) and L is the height of the part of the measurement branch filled with nitrogen (Fig 1).

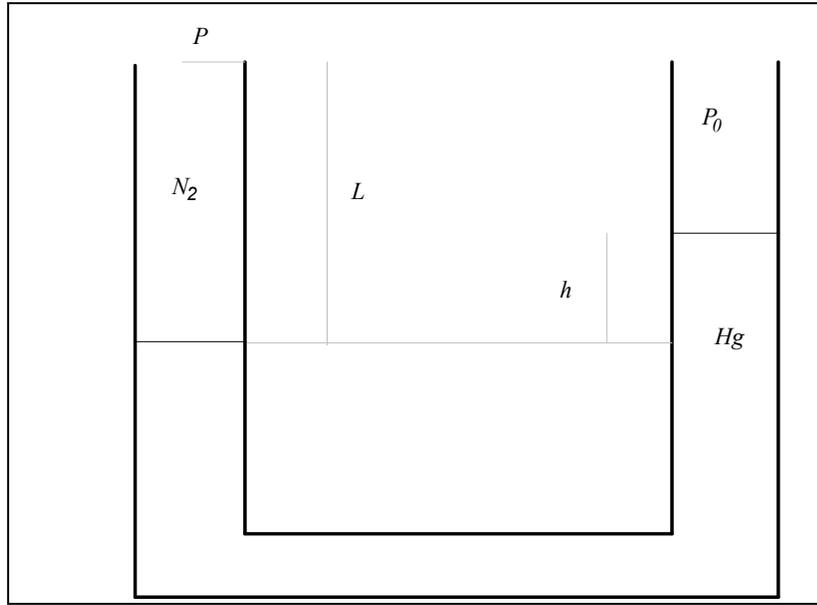


Figure 1. View of mercury column measurement principle

1.2 Design and Development

The mercury column is formed by two tubes connected at their lower ends. These tubes are made of stainless steel that ensures no reaction with mercury and reduces the leakage danger. They have a 50 mm-internal diameter in order to reduce the capillary effect and the hysteresis of the mercury meniscus.

The structure which supports the U-tube and the interferometric system will be made of granite, which is stable and stiff enough in order to ensure the verticality and avoid the influence of thermal deformations.

The mercury density must be accurately measured in order to reduce the standard uncertainty. The mercury density can be expressed as a function of pressure P and temperature t in the following way [2]:

$$\rho_{Hg} = \frac{\rho_{Hg_0}}{[1 + A \cdot (t - t_0) + B \cdot (t - t_0)^2] \cdot [1 - \chi \cdot (P - P_0)]} \quad (3)$$

where parameters A and B take into account the thermal expansion, χ is the compressibility and ρ_{Hg_0} is the density at pressure P_0 and temperature t_0 .

The mercury purity and its thermal stability are important to ensure the density value and, therefore, the system uncertainty.

In order to ensure a 20°C temperature all over the mercury volume the mercury tubes are immersed in a high stability thermostatic bath with water recirculation. The mercury temperature is measured with platinum resistance sensors placed in the bottom and inside the mercury columns. It is expected that the mercury temperature is controlled with a maximum deviation of 20 mK.

The mercury density that will be used in the project is 13545.85 kg/m³ for 20 °C and 101325 Pa with a relative uncertainty of 10⁻⁶. It has been supplied by Riotinto Mines (Huelva, Spain). It is 99.9999% purity in order to ensure the highest stability.

Nitrogen will be considered a real gas in order to calculate its density. Pressure will be generated by means of pressure balances in the measurement branch and also in the reference branch for differential mode.

When measuring absolute pressure, reference pressure should be very low but it cannot be below 0.17 Pa (vapour pressure of mercury) because the mercury vapour would be taken out of the column. Therefore, the reference pressure must be higher than this limit and will be set at approximately 0.25 Pa. This value will be measured by means of a CDG and will be controlled by means of a mass flow controller and a turbomolecular pump.

Local gravity has been determined by means of absolute gravimetry techniques as well as its vertical gradient that oscillates between -2.5·10⁻⁶ s⁻² and -2.9·10⁻⁶ s⁻². The gravity standard uncertainty is better than 2·10⁻⁷ m/s².

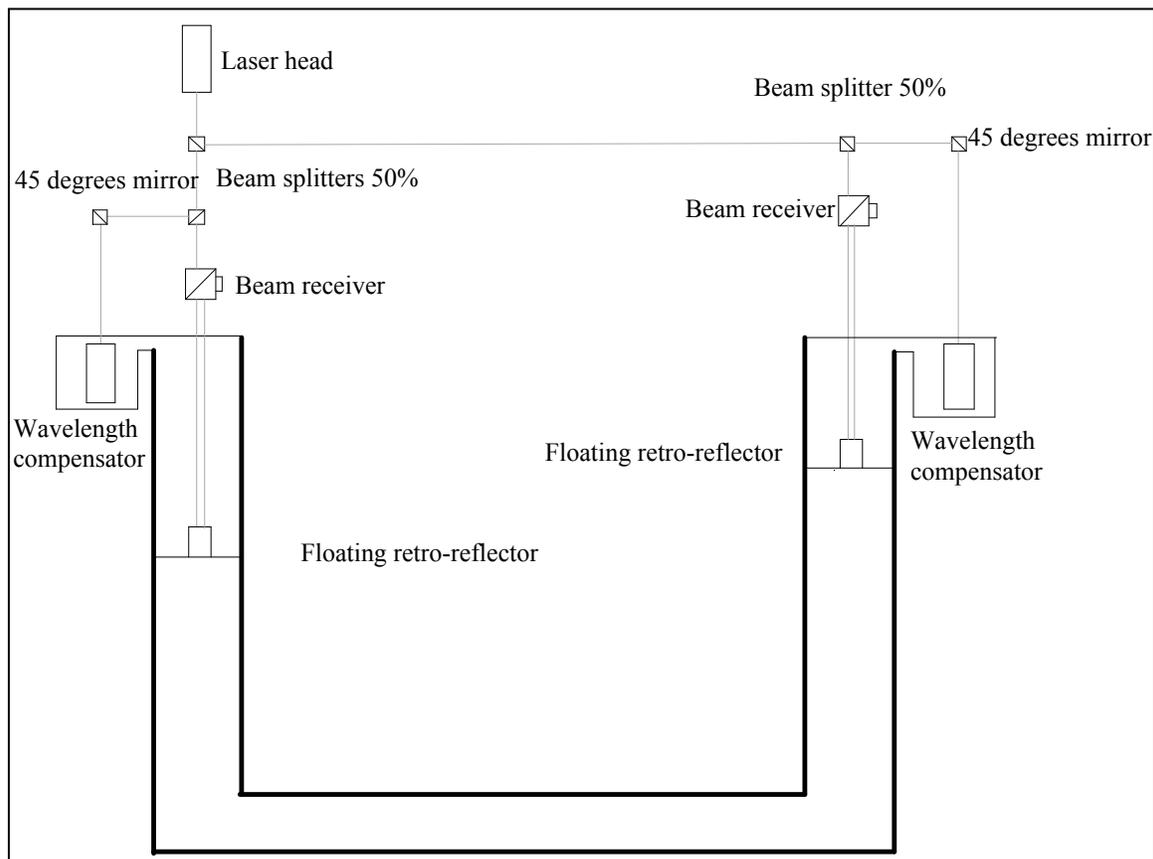


Figure 2. Mercury column interferometric system.

The height differences between mercury levels in reference conditions (both branches have the same pressure) and measurement conditions are measured by means of a helium–neon laser interferometric system (fig. 2), with an accuracy of ± 4 nm. This system has a laser head, three beam splitters, two 45° mirrors, two retro-reflectors (installed on the mercury column surfaces floats) and two beam receivers. It also has two zerodur wavelength compensators to compensate the refraction index (as this index is unknown because it is a mixture of nitrogen and mercury vapour at different pressures, so that Edlen or other equations cannot be used). The floats have been specially designed to be partially sunk in order to avoid capillary and hysteresis effects. It has three contacts with the column in order to centre the float.

One of the first steps has been the vibrational characterization of the laboratory. The study shows there is almost no external vibration so that it is a perfect place for the column, nevertheless a special foundation will be available.

1.3 Expected Results

The main contributions for the uncertainty are caused by temperature, mercury purity and heights measurements (cosine errors, retro-reflector errors, float vibrations and menisci effects). An expanded standard deviation of less than $4 \cdot 10^{-6}$ is expected.

2. Force Balance Piston Gauge

2.1 Description

It is a pressure standard designed to cover the range from 1 Pa to 15 kPa. The instrument operates on the piston gauge principle. However, the force resulting from pressure on the piston is measured by a forced balanced load cell rather than balanced against masses subjected to the acceleration due to gravity.

The FPG [3] uses a 35 mm diameter, tungsten carbide piston cylinder designed with a pressure to mass coefficient 10 kPa /kg. The cylinder has a conical shape on its interior surface to generate a centring force on the piston when a lubrication pressure is applied in the annular gap through the centre of the cylinder. The non-rotating piston is connected to the load cell by a multi degree of freedom mechanical assembly located at its centre of gravity. This allows the piston to centre itself in the cylinder under the influence of the lubricating pressure. As a result, the piston is perfectly mobile, making no contact with the cylinder.

The piston-cylinder resides in the mounting post that provides upper and lower measurement chambers equipped with high and low pressure connections at the two sides of the piston-cylinder. The high pressure port is the measurement port of the FPG. The low pressure port is considered the reference port of the system. When the port is exposed to atmosphere, the FPG will read gauge pressure. If the port is exposed to vacuum, the FPG will measure absolute differential pressure. The measured value of the low pressure port must be applied to FPG differential pressure to get an absolute pressure. This value is measured with a capacitance diaphragm gauge.

The gauge pressure is

$$P_G = \Delta P - (\rho_{high} - \rho_{low}) \cdot g_l \cdot H \quad (3)$$

and the absolute pressure is

$$P_A = \Delta P + P_{ref} - \rho_{high} \cdot g_l \cdot H \quad (4)$$

with ρ_{high} density high side, ρ_{low} density low side, g_l local gravity, P_{ref} reference pressure, H test to FPG reference height and

$$\Delta P = \frac{K_{cal} \cdot (N + \delta N_1 + \delta N_2 + \delta N_3)}{A_{(20)} \cdot [1 + (\alpha_p + \alpha_c) \cdot (t - 20)]} \quad (5)$$

with K_{cal} calibration coefficient, N counts at a specific point, δN_1 air buoyancy correction, δN_2 drag correction, δN_3 reference pressure buoyancy correction, $A_{(20)}$ effective area at 20°C, α_p thermal expansion piston coefficient, α_c thermal expansion cylinder coefficient and t temperature in Celsius.

2.2 Expected results

At the moment we are testing its performance. In the near future we will be able to compare it with piston cylinder assemblies with lower uncertainty so that an expanded uncertainty of less than $40 \cdot 10^{-6}$ is expected.

3. Static Expansion System

3.1 Introduction

In order to provide traceability in the medium and high vacuum ranges CEM is developing a static expansion system. This standard will work from 10^{-4} Pa to 103 Pa.

Static expansion systems are one of the most accurate primary standards in the high and medium vacuum range. These systems are based on Boyle's law taking into account real gas conditions. In the simplest case gas is injected into an evacuated volume v and the pressure p_i is measured. After the measurement of the initial pressure p_i , the gas is expanded into an evacuated calibration volume V . The calibration pressure p_{cal} is given by the following expression,

$$p_{cal} = p_i \cdot \left(\frac{v}{V + v} \right) \cdot \left(1 - \frac{B \cdot p_i}{R_m \cdot T_i} \right) \cdot \left(\frac{T_V}{T_v} \right) \quad (6)$$

where T_i and T_f are the gas temperatures before and after the expansion and the term $1 - (B \cdot p_i / R_m \cdot T_i)$ is the correction for real gases with the molar gas constant R_m . The expansion ratio $f = v/(V + v)$ must be carefully determined. For each value of the calibration pressure the inlet pressure p_i and temperatures have to be measured.

3.2 Design

The new CEM static expansion system will consist of two big and three small volumes separated from each other by valves. Nominally, they will have the following volumes: v_1 and $v_2 \approx 1$ l, V_1 and $V_2 \approx 100$ l, $v_3 \approx 0.5$ l. The main characteristic of the volume configuration is the fact that all vessels are interconnected to each other by means of a manifold (fig. 3).

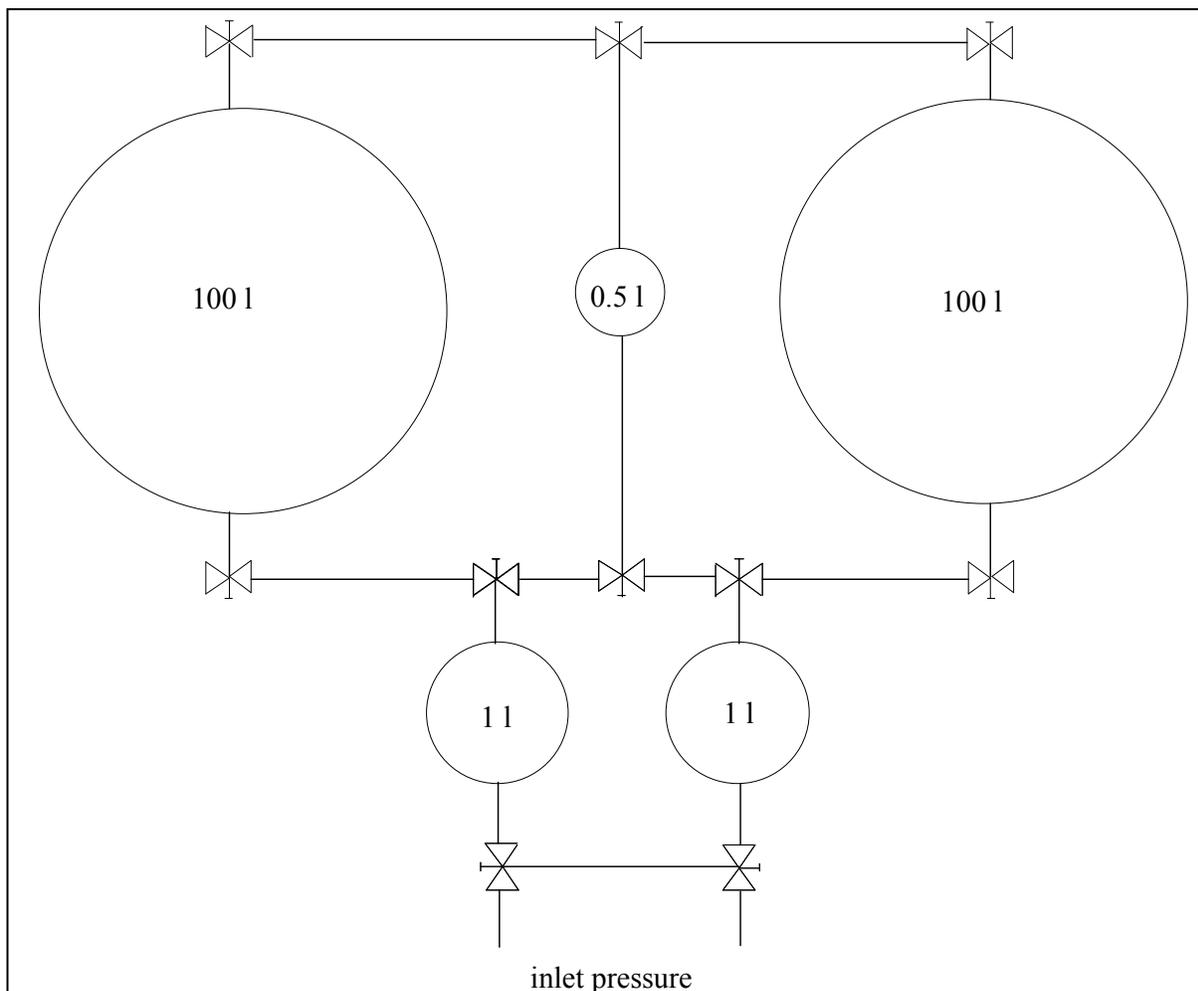


Figure 3. Static expansion system volume configuration

The system will be evacuated by means of two 300 l/s-turbomolecular pumps connected to the large vessels. Both of them will be connected to a primary dry pump. The whole apparatus will be built using the UHV technique with metal seals. The system can be baked up to 300°C. All valves will be manually operated and will be mounted with their seat side towards the vessel.

The initial pressure will be generated by an absolute pressure balance which is able to generate from $1.3 \cdot 10^5$ Pa to 103 Pa with low uncertainty. The final pressure will be measured by a newly calibrated vibrating cylinder gauge. Base pressure and outgassing will be measured in the big vessels by means of calibrated

ionization gauges and any possible leak will be detected by means of a mass spectrometer.

In order to control temperature effects 22 calibrated PT100 sensors will be located on the vessels to correct for temperature differences between the vessels and temperature changes over time (temperature drifts). The readings from temperature sensors will be monitored by RS232 interfaces.

3.3 Generation of Calibration Pressures

The calibration pressure can be generated by various expansion schemes. In a single-step expansion, the gas is expanded from v_1 into $v_1 + V_1$ (or v_2 into $v_2 + V_2$), whereby a reduction of the initial pressure by a factor of about 10^{-2} is obtained. In a two-step expansion the gas is expanded from v_1 into $v_1 + V_1 + v_3$ (or v_2 into $v_2 + V_2 + v_3$), then V_1 (or V_2) valve is closed and again the gas is expanded from v_3 into $v_3 + V_2$ (or into $v_3 + V_1$). As a consequence an overall pressure reduction of less than 10^{-4} is obtained. For a three-step expansion we will start with a two-step expansion case and then V_1 (or V_2) is evacuated and the gas is expanded from v_3 into $v_3 + V_1$ (or v_3 into $v_3 + V_2$) once more obtaining an overall reduction of less than 10^{-6} . In general the pressure will be generated by the following expression,

$$p_{cal} = p_i \cdot R \cdot \left(1 - \frac{B \cdot p_i}{R_m \cdot T_{vi}} \right) \quad (7)$$

where $i = 1$ or 2 depending on the initial volume for the expansion, v_1 or v_2 . For a single step expansion,

$$R = \left(\frac{v_1}{V_1 + v_1} \right) \cdot \left(\frac{T_{V1}}{T_{v1}} \right) \quad \text{or} \quad R = \left(\frac{v_2}{V_2 + v_2} \right) \cdot \left(\frac{T_{V2}}{T_{v2}} \right) \quad (8)$$

For a two-step expansion,

$$R = \left(\frac{v_1}{V_1 + v_1 + v_3} \right) \cdot \left(\frac{T_{V1}}{T_{v1}} \right) \cdot \left(\frac{v_3}{V_2 + v_3} \right) \cdot \left(\frac{T_{V2}}{T_{v3}} \right) \quad \text{or} \quad (9)$$

$$R = \left(\frac{v_2}{V_2 + v_2 + v_3} \right) \cdot \left(\frac{T_{V2}}{T_{v2}} \right) \cdot \left(\frac{v_3}{V_1 + v_3} \right) \cdot \left(\frac{T_{V1}}{T_{v3}} \right)$$

For a three-step expansion,

$$R = \left(\frac{v_1}{V_1 + v_1 + v_3} \right) \cdot \left(\frac{T_{V1}}{T_{v1}} \right) \cdot \left(\frac{v_3}{V_2 + v_3} \right) \cdot \left(\frac{T_{V2}}{T_{v3}} \right) \cdot \left(\frac{v_3}{V_1 + v_3} \right) \cdot \left(\frac{T'_{V1}}{T_{v3}} \right) \quad (10)$$

or

$$R = \left(\frac{v_2}{V_2 + v_2 + v_3} \right) \cdot \left(\frac{T_{V2}}{T_{v2}} \right) \cdot \left(\frac{v_3}{V_1 + v_3} \right) \cdot \left(\frac{T_{V1}}{T_{v3}} \right) \cdot \left(\frac{v_3}{V_2 + v_3} \right) \cdot \left(\frac{T'_{V2}}{T_{v3}} \right)$$

where T' means after the second expansion.

In this system any expansion can be done in two different ways which a way to ensure everything has been done correctly.

3.4 Expansion Ratio Determination

As it can be seen there are several expansion ratios to be determined:

$$\begin{aligned}
 f_1 &= \left(\frac{v_1}{V_1 + v_1} \right) & f_2 &= \left(\frac{v_2}{V_2 + v_2} \right) & (11) \\
 f_3 &= \left(\frac{v_1}{V_1 + v_1 + v_3} \right) & f_4 &= \left(\frac{v_2}{V_2 + v_2 + v_3} \right) \\
 f_5 &= \left(\frac{v_3}{V_1 + v_3} \right) & f_6 &= \left(\frac{v_3}{V_2 + v_3} \right)
 \end{aligned}$$

These expansion ratios will be determined by gas accumulation and SRG methods [4]. The first step will be the calibration of v_1 or v_2 gravimetrically with distilled water. This is necessary in order to know the volume of any gauge we connect to our system. The gauge volume, v_g will be obtained by means of gas expansions. No more accurate method is necessary because this volume is always going to be a small correction. The expansion ratios f_j will be corrected in the following way,

$$f_{j,corr} = \frac{I}{I/f_j - v_g/v_i} \quad (12)$$

where v_i is the initial expansion volume.

In the gas accumulation method initial pressure will be generated in the initial expansion volume by an absolute pressure balance. The final pressure will be measured by a newly calibrated vibrating cylinder gauge connected to the manifold. The initial pressure will be approximately 105 Pa and after 25 expansions a final pressure of 2.104 Pa will be obtained. The vessels temperature will be carefully measured.

In the SRG method a spinning rotor gauge will be connected to the manifold. The initial pressure will be less than 10-1 Pa in order to work in the linear range of the spinning rotor gauge. After each expansion the residual deceleration rate will be determined in order to correct the previous measured deceleration rates before and after the expansion.

The expected uncertainty will be less than 0.05% and temperature corrections will be taken into account for both methods.

3.5 Expected Results

The main effects to be taken into account will be the initial pressure measurements, temperature effects (different temperatures between vessels, drifts, temperature gradients in the large vessels), expansion ratio determination and the virial coefficient. Expanded uncertainties less than 0.15% for a single

expansion calibration, 0.2% for a two-step expansion calibration and 0.25% for a three-step expansion calibration are expected.

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